# On-Site Laboratory Assessment Report (SDWA)

## Radiochemistry
(FINAL 5/18/11)

## Report

**Energy Laboratories, Inc.**
**2393 Salt Creek Highway (82601)**
**P.O. Box 3258**
**Casper, WY 82602**

**Audit ID: 041211_Energy Labs**
**April 12–April 14, 2011**

**Prepared by:**
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**Prepared in Support of**
**EPA Work Assignment 0-15 under EPA Contract No. EP-C-10-060**

**Inspected by:**
[redacted] **CSC**

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# 1.0 Introduction

On April 12 – April 14, 2011, an on-site radiochemistry evaluation was conducted of the Energy Laboratories, Inc. (ELI). The laboratory is located at 2393 Salt Creek Highway, Casper, WY 82601. The purpose of this inspection was to determine the capability of the laboratory to perform its mission as it relates to the Safe Drinking Water Act (SDWA) program and the analyses of drinking water samples for radiochemistry analytes as performed by ELI.

The inspection of radiochemistry was conducted by [redacted], Senior Radiochemist, Computer Sciences Corporation, under contract to the U.S. Environmental Protection Agency's (EPA's) Office of Ground Water and Drinking Water. [redacted], Technical Support Center, Standards and Risk Management Division/Office of Water, US EPA Headquarters, MS-140, 26 W. Martin Luther King Dr., Cincinnati, OH 45268 and [redacted], SDWA Laboratory Certification Manager, US EPA Region VIII, 16194 West 45th Drive, Mail Code: 8TMS-L, Golden, CO 80403, held the opening and closing sessions by teleconference.

References used in this report include the Manual for Certification of Laboratories Analyzing Drinking Water, 5th Edition (2005, U.S. EPA), hereafter referred to as the Certification Manual (CM), and American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories, ANSI N42.23-1996, and American National Standard Calibration and Usage of Alpha/Beta Proportional Counters, ANSI N42.25-1997, hereafter referred to as ANSI.

This assessment included: review of standard operating procedures (SOPs); review of the Quality System (Quality Assurance Manual [a.k.a. Quality Assurance Plan]); interviews; and data review (raw data to final results for Method Detection Limit [MDL] studies, demonstration of performance/capability studies, proficiency testing [PT] analyses, and SDWA compliance data for July 2008 through March 2011).

**Findings (F),** for EPA Region VIII SDWA Laboratory assessments, are defined as factual, objective statements which provide evidence of non-conformance with any of the following:

- the Agency's mandatory methods for the applicable programs the laboratory supports (SDWA);
- official Agency mandates and policies, e.g., 40 CFR 141.25, CFR holding times and preservation requirements, and the CM; and
- the laboratory's Quality Assurance/Quality Control (QA/QC) Manual, QA/QC procedures, and SOPs; and documented practices that adversely affect the quality of the data (e.g., supported with actual data from case files).

**Recommendations (R)** will also be contained in the Laboratory Assessment report. These are <u>not</u> findings, but are the technical opinions of the auditor (or assessment team) that are offered to further improve the laboratory's quality system. They are <u>not</u> requirements.

In addition, **General Comments** may also be included, which are a partial listing of innovative or exceptional items the assessment team wanted to highlight (not intended to be an all inclusive listing).

Only findings need to be addressed in the corrective action plan by the laboratory within 30 days of the receipt of the assessment report.

The report uses the the audit used the Audit Program – Checklist 1, Data Quality for Radiochemical Analysis, Revision 5, November 29, 2010, as the evidentiary documentation of the audit. For full details of the audit along with the findings and recommendations, please refer to the accompanying Audit Checklist.

## 2.0 Personnel

The laboratory was represented by [redacted], Branch Manager; [redacted], Quality Assurance Manager; [redacted], QA; [redacted], Technical Director; [redacted], Radiochemistry Supervisor; [redacted], Radiochemistry Quality Assurance/Quality Control; [redacted], Radiochemistry Technician; [redacted], Radiochemistry Technician; [redacted], Radiochemistry Technician; and [redacted], Radiochemistry Technician.

## 3.0 General Comments

Numerous Quality Control protocols were observed during this on-site inspection, and the laboratory has a Quality Assurance Program Plan. Much data and information were requested prior to the inspection (pre-survey package), and the laboratory was helpful in supplying this information.

The laboratory has a very good sample log in and tracking program/process.

The laboratory has a very good standards traceability process.

## 4.0 Proficiency Testing (PT) Samples

The laboratory has an acceptable PT record.

## 5.0 Analytical Method References

The list of parameters in Section 6.0 was assessed during this inspection with the associated methodology cited as follows:

| | |
|---|---|
| (EPA80) - | Prescribed Procedures for Measurement of Radioactivity in Drinking Water, August 1980, EPA-600/4-80-032. |
| (EPA87) - | Radiochemistry Procedures Manual, December 1987, EPA 520/5-84-006 |
| (CM) - | Manual for the Certification of Laboratories Analyzing Drinking Water, January 2005, EPA 815-R-05-004. |
| (CM1) | Supplement 1 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water, June 2008, EPA 815-F-08-006. |

## 6.0 ELI Analytical Methods for Radiochemistry Information

| Parameter | Method |
|---|---|
| Gross Alpha and Beta | Evaporation (EPA80, 900.0) |
| Radium-226 | Radiochemical (EPA80, 903.0) |
| Radium-228 | Radiochemical (EPA87, Ra-05) |
| Strontium-90 | Radiochemical (EPA80, 905.0) |

| | |
|---|---|
| Tritium | Radiochemical (EPA80, 906.0) |
| Uranium | Radiochemical (EPA80, 908.0) |
| Gamma Emitters | Gamma Spectroscopy (EPA80, 901.1) |

# 7.0 Audit Observations

## 7.1 Personnel

### 7.1.1 Laboratory Management

The laboratory has sufficiently qualified management to meet the requirements of the CM.

### 7.1.2 Laboratory Staff

The auditors found the staff to be experienced and knowledgeable in the required aspects of radiochemistry principles, analyses, and data handling. The laboratory staffing generally satisfy the requirements in Chapter III and Chapter VI, Section 1, of the CM.

## 7.2 Laboratory Facilities

The laboratory has provisions for maintaining the security of samples. The laboratory is roomy and has adequate lighting at the bench top. Exhaust hoods were in place and appropriate to meet requirements for performing radiochemistry procedures. Hood velocities are monitored by the laboratory. Instruments are grounded, and uninterruptible power supplies are available to all instruments. Chemicals are of appropriate grade. Glassware cleaning follows specific requirements in the methods or general requirements in the CM. The laboratory's chemical hygiene plan is adequate to identify and provided for disposal of chemical wastes produced by the laboratory.

Provisions are not made to store high dose rate samples away from drinking water samples. Standards and reference materials are stored away from samples in a separate area of the laboratory.

The radiation protection and radioactive waste programs are appropriate for federal and State regulations with exceptions listed below.

## 7.3 Laboratory Equipment and Instrumentation

Operation of the Gas-flow Proportional, Liquid Scintillation, and Gamma Spectroscopy counting systems generally met the CM requirements with exceptions listed below.

## 7.4 General Laboratory Practices

Laboratory operational practices generally meet the CM requirements.

## 7.5 Analytical Methodology

Standards' National Institute of Standards and Technology (NIST)-traceable preparations meet CM requirements.

Approved radioanalytical methods cited in 40 CFR 141.25(a) and (b), Table VI-1 are modified for use in the radiochemistry laboratory without the modifications detailed in the SOPs. The laboratory does not have alternate test procedure (ATP) approvals, in accordance with 40 CFR 141.27, for these procedure modifications. Details of these modifications are noted below.

### 7.6 Sample Collection, Handling, and Preservation

Samples are submitted to the laboratory, for analysis, from various clients from across the United States. Handling, compositing, and preservation of samples do not meet CM requirements in all cases, and these deficiencies are detailed below.

### 7.7 Quality Assurance

The laboratory has a written, separately prepared QA manual (QAM [a.k.a. QA plan]) and generally met CM requirements, with exceptions noted below. Laboratory safety plans were thorough.

### 7.8 Records and Data Reporting

Records and Data Reporting generally met CM requirements, with exceptions noted below.

## 8.0 Audit Findings

### 8.1 Quality Systems - Laboratory Quality Assurance Plan

#### 8.1.1 Requirement – CM Ch. III, Section 11.6; Checklist Items 2.8 and 6.1

The QA Plan addresses instrument calibration procedures (may reference SOP) and describes:

- specific type of calibration used for each method and frequency of use;
- calibration standards' source, age, storage, labeling;
- data comparability checks; and
- control charts and for radiochemistry, report counting errors with their confidence levels.

**Finding** – The laboratory's QAM, Chapter 7, requires laboratory SOPs to detail the sequence of operations involved in instrument startup, calibration, analyzing, shutting down, and routine maintenance. The acceptance criteria for the calibration curves are listed in the individual methods. Calibration standards are routinely compared against second source calibration standards to verify accuracy. These second source standard results must fall within an established range, as described by the SOP, to be accepted. The laboratory's radiochemistry SOPs are extremely limited in scope as per this QAM requirement. The SOPs do not detail the acceptance criteria for calibrations.

#### 8.1.2 Requirement – CM Ch. III, Section 11.8; Checklist Item 2.10

The QA Plan addresses data reduction, validation, reporting and verification (may reference SOP) and describes:

- data reduction process (e.g., method of conversion of raw data to mg/L, picocuries/L, etc.);
- data validation process;
- reporting procedures, including format;
- data verification process;
- reporting of counting uncertainties and confidence levels; and
- procedure for data corrections.

**Finding** – Data reduction, validation, verification process, and reporting are addressed in laboratory's QAM; Chapter 8, states:

> The data recorded on the draft report is validated with several steps. The analyst who submits the report checks all the values reported for omissions and accuracy. Elements of this review also evaluate all instrument and method QC results. Automated data management programs are designed with an interactive step allowing data review by the analyst. Results to be reported are approved by the analyst. **The reported result and associated QC data is reviewed by the supervisor. Supervisors review the suitability of the data according to project and method performance specifications.** Analyses results for each requested parameter are evaluated against other requested parameters, project specifications, other samples within the set, historical files associated with the project/client, and any other information provided with the sample. **Supervisors initial all validated sample results.** *[emphasis added]*

A random data package (Batch ID GrAB-105, Run Date 3/3/2011) for gross alpha/beta, by EPA Method 900.0 was selected for validation during the audit. The auditor requested the Radiochemistry Manager to demonstrate the reported value (162.7 ± 10.3 pCi/liter gross alpha) for sample I.D. C11030083-001B was calculated correctly. Several efficiency absorption curves were provided to try to establish which curve was used to determine the counting efficiency for the sample. It was discovered that the laboratory takes multiple efficiency curves and determines an average efficiency from the curves, and applies that efficiency to all detectors in the counting system. The laboratory's approach is inappropriate because it does not have the individual efficiency response for the specific detector that the compliance sample was counted on. Additionally, the Radiochemistry Manager could not provide the efficiency that was used to calculate the compliance sample activity. Several phone calls to the corporate office (where the laboratory information management system [LIMS] is located), and a couple hours later, a secondary calculation sheet was provided explaining the derivation of the calculation for efficiency. Based on the auditor's experience, an efficiency of approximately 10.27 % at 50 mg solids absorption is somewhat accurate and the reported result is likely to be correct (**with the exception of that the laboratory uses average backgrounds to calculate activity [See Audit Finding 8.5.2 below]**). However, the validation, during the audit, uncovered that data being produced, by the laboratory, are not being validated as required by the CM and the laboratory's QAM.

Additionally, the gross alpha results exceeded the 40 CFR 141.66(c) maximum contaminant level (MCL) for gross alpha particle activity (excluding radon and uranium). The MCL for gross alpha particle activity (including radium-226 but excluding radon and uranium) is 15 pCi/L; a radium-226 analysis was performed (Batch I.D. RA226-5214, Sample I.D. 11030083-001B and 11030083-001BDUP) with radium-226 results of 18.70 ± 0.99 and 12.25 ± 0.80 pCi/liter respectively. The **relative percent difference for these duplicates was 42%**, which exceeds the CM Ch. VI, Section 7.7.1 requirements that precision must be ≤ 20%. If precision assessments exceed their limits (relative percent difference [RPD] or replicate error ratio [RER]), calculations and procedures are examined and samples recounted and re-evaluated. If the precision assessments are still unsatisfactory after the samples are recounted, then all sample results in the preparation batch are to be reported with a qualifier to indicate the measurement has questionable precision. If the client requires unqualified results, then all the samples in the sample preparation batch are discarded and then re-measured using new aliquots of the sample if holding times allow and sufficient volume is available. Otherwise, the water is resampled and reanalyzed. The samples were flagged during the data validation but not recounted to verify the precision. Additional QC issues exist with the radium-226 batch, cited above, with the laboratory fortified blank (LFB) and (a.k.a. laboratory control sample [LCS]) recovery. The LFB recovery, for Batch I.D. RA226-

5214, was 119% of the known added amount of radium-226 spike. CM Ch. VI, Section 7.7.2 requires that LFB recoveries be within ± 10% of the known added amount for radium-226. The laboratory's limit, which is not compliant with CM Ch. VI, Section 7.7.2, is ± 15%. Clearly, the LFB recovery exceed the CM and the laboratory's' requirements.

Also. the CM Ch. VI, Section 7.7.3, states:

> If LFB assessments exceed their limits, calculations and procedures should be examined and samples recounted. If the LFB assessment is still unsatisfactory after the samples are recounted, the batch must be considered contaminated. All samples in the sample preparation batch must then be discarded. After the source of contamination is identified and addressed, all the samples in the discarded sample preparation batch must then be re-measured using new aliquots of the sample if hold times and sufficient volume is available.

Additional requirements are also found in Section 7.7.2:

> When MS [matrix spike] assessments of accuracy exceed their calculated control limits, calculations and procedures should be [are] examined and samples recounted. If the assessment is still unsatisfactory after the samples are recounted, then all sample results in the batch should be flagged as possibly biased low or high (as the result indicates) due to matrix effects. If the LFB assessment of accuracy independent of matrix effects is also unsatisfactory in the same preparation batch (see below [a reference to Section 7.7.3]), then all the samples in the discarded sample preparation batch are remeasured using new aliquots if holding times allow and sufficient volume is available.

The batch was not recounted or discarded and the samples were not reprepared and recounted.

The auditor considers the laboratory's data review, verification, and validation process broken and with total disregard to the requirements of the CM and the laboratory's own QAM. **The auditor considers this finding an extremely serious finding.** The auditor recommends that that laboratory cease analyzing drinking water compliance samples until **all compliance sample data**, which has been produced by the laboratory for the last 3 years, can be examined, verified, and validated as being compliant with the CM requirements and the laboratory's QAM requirements.

### 8.1.3 Requirement – CM Ch. III, Section 11.9; Checklist Item 2.11

The QA Plan addresses types of QC checks and the frequency of their use (may reference SOP). Parameters for radiochemistry should include or reference:

- instrument performance check standards;
- frequency and acceptability of method detection limit (MDL) calculations;
- frequency and acceptability of demonstration of low level capability;
- calibration, internal and surrogate standards;
- laboratory reagent blanks, field reagent blanks and trip blanks;
- field and laboratory matrix replicates;
- QC and proficiency testing samples;
- laboratory fortified blanks and laboratory fortified sample matrix replicates;
- initial demonstration of method capability
- use of control charts; and

- qualitative identification/confirmation of contaminants.

**Finding** – The laboratory's QAM does address types of QC checks; however the radiochemistry QC check limits are set at various conflicting values (SOPs vs. LIMS) ± 20% in some of the SOPs and ± 30% for all radioanalytes except radioactive strontium that is set at ± 50%. In short, some SOP values do not match up with the LIMS values. The LIMS values are the values used to generate the analytical reports. These limits are in direct conflict with the CM Ch. VI, Sections 7.7.2 and 7.7.3, as follows:

> The percent recovery [for MS samples] should be within the control limits calculated from previous MS measurements, which should ideally be ± 20 percent of the amount of activity added to the MS sample. For gross alpha particle activity, gross beta particle activity measurements, and Ra-228 methods of analyses, where experience has shown lower accuracies can be expected, control limits calculated from previous MS results should be within ± 30 percent of the amount of activity added to the MS for the accuracy relative to the sample matrices to be considered acceptable....
>
> LFB accuracy is assessed using the percent recovery of the known activity of radioanalyte added to the sample. The percent recovery should be within the control limits calculated from previous results, which should ideally be within ± 10 percent of the amount of activity added to the LFB sample. For gross alpha particle activity, gross beta particle activity, and Ra-228 measurements, where lower accuracies can be expected, the calculated control limits should not exceed ± 20 percent recovery of the amount of activity added to it.

**This is a repeat finding from the radiochemistry audit performed in July 2008.**

### 8.1.4 Requirement – CM Ch. III, Section 11.10; Checklist Item 2.12

The QA Plan addresses schedules of internal and external system and data quality audits and inter laboratory comparisons (may reference SOP). The audit program includes:

- independent assessments by technically qualified personnel;
- maintenance of an audit schedule;
- audit procedures;
- standard formats for reporting findings to laboratory management; and
- methods for implementing and verifying corrective actions.

**Finding** – The laboratory's QAM, Chapter 2, details the requirements for annual internal audits of the laboratory; however, there have been no internal audits of the radiochemistry laboratory in more than 2 years.

### 8.1.5 Requirement – CM Ch. III, Section 11.11; Checklist Item 2.13

The QA Plan addresses preventive maintenance procedures and schedules and describes:

- the location of instrument manuals and schedules and documentation of routine equipment maintenance;
- the availability of instrument spare parts in the laboratory; and
- a list of any maintenance contracts in place.

**Finding** – The laboratory's QAM does not address the availability of instrument spare parts in the laboratory or list of any maintenance contracts in place.

## 8.2 Initial and Ongoing Demonstrations of Proficiency for Analysts and Technicians

### 8.2.1 Requirement – CM Ch. VI, Section 1.5; Checklist Item 3.3

Demonstrations of Proficiency are performed by preparing and measuring a sample set of at least four reagent blanks and four laboratory fortified blanks that have the radioanalyte of interest added to them at a known concentration appropriate for the relevant method. Performance standards are as follows:

- the activity level added to the laboratory fortified blanks should be between the radioanalyte MCL and its required detection limit;
- the mean recoveries and the standard deviation of the recoveries of the replicate measurements should be consistent with the requirements for accuracy and precision described in MCLADW Ch. VI, Section 7.7;
- reagent blank measurements have a mean result below the method detection limit for each analyte measured; and
- all Initial and Ongoing Demonstrations of Proficiency, along with all records related to methods training and external training relevant to laboratory operations, should be recorded in a training file specific for each analyst and technician.

**Finding** – The laboratory's LFB acceptance criteria for LFBs were not consistent (± 30% for all analytes except radioactive strontium that was set to ± 50%) with the LFB recoveries cited in the CM Ch. VI, Section 7.7.3:

> LFB accuracy is assessed using the percent recovery of the known activity of radioanalyte added to the sample. The percent recovery should be within the control limits calculated from previous results, which should ideally be within ± 10 percent of the amount of activity added to the LFB sample. For gross alpha particle activity, gross beta particle activity, and Ra-228 measurements, where lower accuracies can be expected, the calculated control limits should not exceed ± 20 percent recovery of the amount of activity added to it.

Because the laboratory used the wrong acceptance criteria in assessing demonstrations of proficiencies (a.k.a. demonstrations of capabilities [DOC]), all DOCs performed by the laboratory analyst must be re-evaluated against the LFB acceptance criteria in the CM Ch. VI, Section 7.7.3. DOCs that fail this acceptance must be re-performed and the analyst must not perform compliance sample method analysis until a successful DOC is completed for the specific method.

## 8.3 Laboratory Facilities

### 8.3.1 Requirement – CM Ch. VI, Section 2.1 (Check List Item 4.3)

The laboratory must have provisions for the proper disposal of chemical and radiological wastes, including liquid scintillation cocktail mixtures.

**Finding:** The laboratory is in the process of developing a radioactive waste tracking system; however, the laboratory does not track how much radioactive waste is discharged through the laboratory's sanitary sewer system. The laboratory cannot demonstrate that they are in compliance with their radioactive materials license.

### 8.3.2 Requirement – CM Ch. VI, Section 2.1 (Check List Items 4.4 and 4.5)

Analytical and sample storage areas must be isolated from all potential sources of contamination.

**Finding:** The laboratory does survey samples, when received, to determine each samples radiation dose rate; however, it was observed that samples marked as radioactive were being stored (sitting side-by-side) with samples that were not radioactive. The laboratory must physically separate and store, in a secured location, known radioactive samples away from other drinking water samples.

## 8.4 Laboratory Equipment and Instrumentation (General)

### 8.4.1 Requirement – CM Ch. VI, Section 2.1 (Check List Item 4.3)

The laboratory must have provisions for the proper disposal of chemical and radiological wastes, including liquid scintillation cocktail mixtures.

**Finding:** The laboratory is in the process of developing a radioactive waste tracking system; however, the laboratory does not track how much radioactive waste is discharged through the laboratory's sanitary sewer system. The laboratory cannot demonstrate that they are in compliance with their radioactive materials license.

## 8.5 Gas-flow Proportional Counting System

### 8.5.1 Requirement – CM Ch. VI, Section 3.1.2; Method Specific; ANSI N42.25, Section 5.1; ANSI N42.25, Section 4.3, 6.4 (Check List Items 6.4.5, 6.4.6, and 6.4.8)

Initial calibration solids absorption curves are prepared for alpha, beta self-absorption, and alpha cross-talk:

- the curves consist of points that are well distributed throughout the mass range; and
- self-absorption curves exist for both alpha and beta counting.

Cross-talk determination:

- The sources used for the determination of self absorption and cross-talk curves are of similar isotope content to that of the analytical samples. Th-230 or Am-241 (Method Specific) is used for alpha; Cs-137 or Sr-90/Y-90 (Method Specific) is used for beta.
- Vendor software used to calculate results provides for cross-talk correction; or calculations of sample activity that are performed manually or programmed by the laboratory provide cross-talk correction.

Solids absorption curves are re-verified on a regular basis by:

- re-measuring and generating a new curve; or
- re-verifying the current accuracy of the original curve with at least three of the original solids absorption standards used to produce the solids absorption curve currently in use for data reduction are selected for re-measurement and should span the range of weights used in the original solids absorption curve;
- for the re-verification measurement to be acceptable, the original measurement of the solids standard should occur within the range defined by the uncertainty of the re-verification measurement calculated at the 95% confidence level; also
- performing re-verification of solids absorption curves at least on an annual basis.

**Finding:** The laboratory does not correct for cross-talk when counting alpha and beta analysis simultaneously, as required by EPA Method 900.0, Step 9.2.2. Absorption curves do not exist for each alpha/beta detector in the counting systems. Each detector must have its own absorption curve and not an average curve generated for multiple (16) detectors.

The laboratory's SOP requires re-verification only once every 3 years. The laboratory

verifies only an average efficiency across several detectors and not each independent detector. This practice is unacceptable because it assumes that all of the detectors in the system are identical. Each detector is unique and its individual detector characteristics must be quantified for the measurement of compliance samples. Furthermore; using the average detector response approach does not accurately account of the differences between detectors in the combined standard uncertainty (CSU) error for individual detector analysis of compliance samples.

Documentation of absorption curves was ill-kept and almost impossible to follow. The laboratory must devise a better data system for filing calibration data.

**8.5.2 Requirement – CM Ch. VI, Section 3.1.2 (Check List Item 6.4.11)**

Daily efficiency and background check data is documented, retained, and monitored using control charts or tolerance charts.

**Finding:** Efficiency and background checks are performed daily and documented; however, the laboratory uses an average efficiency and average background (i.e., for all the detector in the counting system) approach, which is unacceptable.

The following alpha background data were used in calculating compliance sample(s) activity in the laboratory's Batch I.D. GrAB-1051, sample I.D. C11030083-001B (positive 5.4 pCi/liter difference):

| Average Background Used | Actual Detector 2B Bkg. | Sample Count Time | Over Subtracted Bkg Counts | Reported Sample Conc. in pCi/L | Corrected Sample Conc. in pCi/L |
|---|---|---|---|---|---|
| 0.127 cpm | 0.0778 cpm | 720 min | 35.4 | 162.7 | 168.1 |

Conversely: If the sample **had been counted** (random selection of detectors when the counting sequence is loaded) on detector 3C, the following **would have been** the correction (negative 10.2 pCi/liter difference):

| Average Background Used | Actual Detector 3C Bkg. | Sample Count Time | Under Subtracted Bkg Counts | Reported Sample Conc. in pCi/L | Corrected Sample Conc. in pCi/L |
|---|---|---|---|---|---|
| 0.127 cpm | 0.2222 cpm | 720 min | 68.5 | 162.7 | 152.5 |

**The differential between these two corrections is based only on the differences between the average background used by the laboratory, and the true background of a specific detectors for one sample.** Depending on what detector any specific compliance sample is counted on, the true reportable result can be off by as much as plus 5.4 pCi/liter or minus 10.2 pCi/liter. This situation is very concerning when evaluating compliance sample data. If a sample, for gross alpha, was analyzed on the laboratory's detector 2B, under the current laboratory practice, and had a calculated analysis result reported as 10 pCi/liter, the true analysis result would be 15.4 pCi/liter, and, in actuality, the gross alpha would exceed the

gross alpha MCL. Additional radium-226 analysis would be required. However, without knowing the true result, the health and safety of the public is at risk. Conversely, if the same sample was counted on detector 3C, the reported concentration would be a minus 0.2 pCi/liter. In reviewing the calculation data for Batch I.D. GrAB-1051, there are eight sample results that are negative values. These samples results must be reviewed and recalculated to ensure the appropriate gross alpha activity was reported.

The laboratory uses 2 different (multi-detector) gas proportional counters to count compliance samples for gross alpha and gross beta by evaporation, gross alpha by co-precipitation, radium-226, radium-228, and radioactive strontium. The laboratory uses the same average background and efficiency approach for both systems and analysis. The laboratory's LIMS is hard coded to perform these calculations. The laboratory must stop using their average background and efficiency approach in calculating compliance sample activity. Drinking water compliance sample MCL violations can easily be biased (high or low) by the current practice. The laboratory must cease analyzing and reporting results for compliance samples, for the above referenced methods, until the actual detector background and efficiency, for each detector in the counting system(s), can be used to calculate compliance sample results. Furthermore; the laboratory must review compliance sample data for the last 3 years and recalculate the true activity for compliance samples analyzed, for all of the above referenced methods, and issue corrected reports.

**8.5.3 Requirement – CM Ch. VI, Section 3.1.2 (Check List Item 6.4.12)**

If instrument control measurements exceed their control limits, the proportional counter is placed out of service until the reason for the change in efficiency or background can be determined and corrected.

**Finding:** Because the laboratory is using the average background and efficiency to determine control limits, they would not know if any one single detector was out of control; thus, take it out of service.

**8.5.4 Requirement – CM Ch. VI, Section 3.1.2 (Check List Item 6.4.14)**

Efficiency calibration is performed annually, and when one or more of the following occur:

- a hardware component has been replaced or repaired;
- changes have been made to the system (sample preparation procedure changed or gas flow adjusted);
- quality or manufacturer of counting gas has changed; or
- instrument performance checks indicate a change in the instrument response.

**Finding:** Absorption efficiency curves are only generated once every 3 years in accordance with the laboratory's SOP; thus this requirement cannot be met.

**8.5.5 Requirement – CM Ch. VI, Section 3.1.2 (Check List Item 6.4.15)**

The alpha and beta background count of the system should be low enough so that the sensitivity of the radioanalysis of water samples will meet the requirement of 40 CFR 141.25(c) within reasonable counting time (not more than 1,000 minutes).

**Finding:** The laboratory's MDL studies for gross alpha and gross beta failed with MDLs equal to 7 and 9 pCi/liter, respectively. The required detection limits are 3 and 4 pCi/liter,

respectively. The laboratory performed the **failed MDL studies on February 17, 2011**; however, the laboratory continued to analyze compliance samples and reporting data (**the laboratory's gross alpha and gross beta analysis of Batch LD. GrAB-1051, was run on March 3, 2011**). The laboratory was unable to comply with the detection limits required in 40 CFR 141.25(c) and thus is in violation of the statute by reporting drinking water compliance sample results. **This is a very serious finding.**

## 8.6 Gamma Spectrometer Systems

### 8.6.1 Requirement – CM Ch. VI, Section 3.1.5 (Check List Item 6.7.1)

The gamma detector system consists of detectors suitable for measuring the gamma isotopes of interest in the range of $\geq 0.06$ to $\leq 2$ MeV, and the efficiency of the detector is adequate to meet the detection limits listed at 40 CFR 141.25(c), and bias and precision requirements.

**Finding:** Inappropriate gamma emitters (e.g. americium-241 [MCL is for alpha emitter] and potassium-40 [non-regulated natural occurring radioisotope]) were selected to perform the laboratory's MDL study. The laboratory must repeat their MDL study for gamma emitters. It is recommended that the radioisotopes used in the national performance testing program be used in the laboratory's repeated MDL study. The following are the required detection limits in pCi/liter: Ba-133 – 152, Cs-134 – 10, Cs-137 – 20, Co-60 – 10, and Zn-65 – 30.

### 8.6.2 Requirement – CM Ch. VI, Section 3.1.5 (Check List Item 6.7.4)

Background measurements are performed on at least a monthly basis, and the background gross gamma activity recorded.

**Finding:** Background measurements are performed on quarterly basis and are not compliant with this requirement.

## 8.7 Analytical Methods – Standard Operating Procedures

### 8.7.1 Requirement – CM Ch. VI, Section 5.1 (Check List Items 7.0, 7.2.1, 7.2.4, 7.2.5, 7.2.6, 7.2.12, 7.2.14, 7.2.15, and 7.2.17)

The approved methods cited at 40 CFR 141.25(a) and (b), Table VI-1, must be used for the analysis of drinking water compliance samples. A laboratory-specific SOP should be written for each method used for measuring regulated radio-analytes in compliance monitoring samples. These SOPs should be consistent with a referenced approved method, and any EPA-approved modifications should be noted.

**Finding:** Several of the laboratory's SOPs are modified and are not consistent with the referenced approved method. The SOP modifications do not have EPA approval in accordance with 40 CFR 141.27. The non-compliant SOPs are as follows:

- The laboratory's SOP for gross alpha and beta by EPA Method 900.0 is modified without being EPA approved through the alternative test procedure (ATP) process. The laboratory was provided with a marked copy of their SOP annotating the deviations from the approved method.
- The laboratory's SOP for radium-226 by EPA Method 903.0 was significantly modified (as a sequential radium-226 and radium-228 method) without being EPA approved through the alternative test procedure (ATP) process. The laboratory was provided with a marked copy of their SOP annotating the deviations from the approved method.
- The laboratory's SOP for radium-228 by EPA Method Ra-05 was significantly modified

without being EPA approved through the alternative test procedure (ATP) process. The laboratory was provided with a marked copy of their SOP annotating the deviations from the approved method. **This is a repeat finding from the audit performed in July 2008.**

- The laboratory's SOP for uranium by EPA Method 908.0 was modified without being EPA approved through the alternative test procedure (ATP) process. The laboratory was provided with a marked copy of their SOP annotating the deviations from the approved method.
- The laboratory's SOP for radioactive strontium by EPA Method 905.0 was modified without being EPA approved through the alternative test procedure (ATP) process. The laboratory was provided with a marked copy of their SOP annotating the deviations from the approved method.
- The laboratory's SOP for gamma emitters by EPA Method 901.1 fails to implement step 9.1.1, which requires that all photo peaks must be identified. The laboratory was provided with a marked copy of their SOP annotating the deviations from the approved method.

## 8.8 Sample Collection, Handling, and Preservation

### 8.8.1 Requirement – CM Ch. VI, Section 6 (Check List Items 8.3, 8.4, and 8.8)

Sample preservatives provided by the laboratory should be screened for radioactive content by lot number prior to their use in the laboratory, and the results documented. Samples preserved with reagents that are not provided by the laboratory are to be accompanied by a radioactive-free field blank sample that is preserved in the same manner as the submitted sample. A sample of the preservative should accompany the composited sample to the laboratory to determine the contribution of radioactivity, if any, from the addition of the preservative.

**Finding:** Preservative reagent lots are tracked when preparing preservation ampoules to be sent with the sample bottles; however, there are several lots of preservative made up in ampoules, and the lot identification is not transcribed to the ampoules. The laboratory does not know what lot of preservative was used in any one particular sample when it is taken, which defeats the purpose of tracking preservative lots. The laboratory does not require samplers, who preserve samples with reagents not provided by the laboratory, to provide a field blank sample in order to assess if any radioactivity has been added, to the compliance drinking water sample, from the preservatives used by the sampler(s). Additionally, the laboratory does not require a sample of the preservative should accompany the composited sample to the laboratory to determine the contribution of radioactivity, if any, from the addition of the preservative.

**This is a repeat finding from the audit performed in July 2008.**

## 8.9 Method Sensitivity Studies

### 8.9.1 Requirement – CM Ch. VI, Section 7.3 (Check List Items 9.3.1, 9.3.2, and 9.3.3)

The laboratory should determine the standard analytical conditions for each method for measuring compliance monitoring samples that can produce detection limits that are equal to or less than those specified in 40 CFR 141.25(c)(1) Table B, and 40 CFR 141.25(c)(2) Table C.

Even though the CM states that sensitivity (MDL) determinations should be consistent with

Appendix C of "Prescribed Procedures for the Measurement of Radioactivity in Drinking Water" (EPA-600/4-80-032), EPA OGWDW has since determined that MDLs must be calculated according to Appendix B to 40 CFR Part 136, "Definition and Procedure for the Determination of the Method Detection Limit," Rev. 1.11.

Once the method standard analytical conditions are determined, the laboratory should institute a monitoring program to ensure that the sensitivity of each method used to analyze compliance monitoring samples does not exceed the detection limits specified in 40 CFR 141.25(c)(1) Table B, and 40 CFR 141.25(c)(2) Table C.

**Finding:** 40 CFR 141.25(c) required MDLs have not been successfully demonstrated for some methods and some MDL studies were inappropriate based on the radioanalyte selected. The laboratory must repeat the following MDL studies:

- Gross Alpha
- Gross Beta
- Radium-226
- Radium-228
- Radioactive Strontium
- Gamma Spectroscopy

Also, once the laboratory decides what approved method they are going to use to analyze Uranium by, they must perform a MDL study.

The laboratory was not able to achieve several detection limits as required in 40 CFR 141.25(c); however, they continue to analyze and report compliance sample results. **This is a very serious finding.**

## 8.10 Proficiency Test (PT) Studies

### 8.10.1 Requirement – CM Ch. III, Section 13.1 (Check List Item 9.4.3)

At least annually, drinking water laboratories certified for chemical contaminants must satisfactorily analyze a PT sample to maintain certification (40 CFR 141.23(k)(3)(i), 141.24(h)(17)(i)(A), and 141.89(a)(1)(i)). PT samples should be analyzed in the same manner as routine samples. Laboratories must acquire the PT sample from a supplier acceptable to the appropriate certification authority.

**Finding:** The laboratory analyzes PT samples multiple times with some RPDs at 45.8% (Ra-226 run 4 times with the following results in pCi/liter: 15.94, 10.0, 15.56, and 14.08). The laboratory reported 15.6 pCi/liter as their PT result (if they had reported the 10.0 pCi/liter result, they would have failed the PT). The laboratory did not have a logical explanation why one result was reported over another. The laboratory must analyze PT samples identically as other compliance samples are analyzed (single analysis per sample).

## 8.11 Sample Measurement Quality Control Requirements

### 8.11.1 Requirement – CM Ch. VI, Section 7.7.1 (Check List Items 9.7.5 through 9.7.7)

An RPD result that exceeds its calculated control limit (which ideally should be 20 percent or less) indicates the precision of the sample preparation batch is questionable, and data reported from these results should be flagged as having questionable precision. Duplicated sample measurement activities that are less than 5 times the radioanalyte's detection limit, and exceed 20 percent RPD when compared to the first measurement for the sample are:

re-evaluated using the two measurement's RER using the formula:

$$RER = |A - B| / SQRT( s_a^2 + s_b^2 ) \leq 2$$

If the RER exceeds 2, it is noted as unacceptable.

If precision assessments exceed their limits (RPD or RER):
Calculations and procedures are examined and samples recounted and re-evaluated.
If the precision assessments are still unsatisfactory after the samples are recounted, then all sample results in the preparation batch are to be reported with a qualifier to indicate the measurement has questionable precision. If the client requires unqualified results, then all the samples in the sample preparation batch are discarded and then re-measured using new aliquots of the sample if holding times allow and sufficient volume is available. Otherwise, the water is resampled and reanalyzed.

**Finding:** Some duplicate acceptance criteria, in the laboratory's SOPs, was set at 25% RPD. Not all of the laboratory's SOPs address the calculation of RER. ***Also see audit finding 8.1.2 for additional RDP non-conformances.***

**8.11.2 Requirement – CM Ch. VI, Section 7.7.1 (Check List Items 9.7.9 , 9.7.11, and 9.7.12)**

MS performance is assessed using the percent recovery of the known activity of radioanalyte added to the sample and should be within:

- ± 20 percent of the amount of activity added; or
- for gross alpha, gross beta, and Ra-228, should be within ± 30 percent of the amount of activity added.

When MS assessments of accuracy exceed their calculated control limits, calculations and procedures should be (are) examined and samples recounted. If the assessment is still unsatisfactory after the samples are recounted, then all sample results in the batch should be flagged as possibly biased low or high (as the result indicates) due to matrix effects. If the LFB assessment of accuracy independent of matrix effects is also unsatisfactory in the same preparation batch, then all the samples in the discarded sample preparation batch are re-measured using new aliquots if holding times allow and sufficient volume is available.

**Finding:** The laboratory LIMS is monitoring all methods at ± 30 percent, with the exception of radioactive strontium, which is set to ± 50 percent. The laboratory needs to change their LIMS to reflect the requirements of this line of inquiry and revise their all SOPs to be consistent with this requirement. ***Also, see audit finding 8.1.3 for further details.*** **This is a repeat finding from the audit performed in July 2008.**

**8.11.3 Requirement – CM Ch. VI, Section 7.7.3 (Check List Items 9.7.13 through 9.7.15)**

Assessing the preparation batch accuracy independent of matrix effects using laboratory fortified blanks (LFB):

- LFBs are prepared using deionized water;
- an LFB shall be prepared with each preparation batch of samples;
- LFBs are processed through the method along with the sample batch; and
- LFBs are assessed using percent recovery calculations and should be within the control limits ± 10 percent of the amount of activity added to the LFB sample.

Except:

- for gross alpha, gross beta, and Ra-228, LFBs should be within ± 20 percent of the amount of activity added

If LFB assessments exceed their limits, calculations and procedures should (are) be examined and samples recounted. If the LFB assessment is still unsatisfactory after the samples are recounted:

- the batch is be considered contaminated;
- all samples in the sample preparation batch are discarded;
- the source of contamination is identified and addressed; and
- new aliquots of the sample(s) in the preparation batch are measured if holding times allow and sufficient volume is available.

**Finding:** The laboratory LIMS is monitoring some methods LFBs at ± 15% and some at ±30%. The laboratory needs to change their LIMS to reflect the requirements of this line of inquiry and revise their SOPs to be consistent with this requirement. ***Also, see audit finding 8.1.3 for further details.*** **This is a repeat finding from the audit performed in July 2008.**

### 8.11.4 Requirement – CM Ch. VI, Section 7.7.3 (Check List Items 9.7.16 and 9.7.17)

When assessing instrument drift during sample measurements, efficiency and background are checked:

- before measuring compliance monitoring samples;
- after measuring compliance monitoring samples; during long analytical batch counting times (greater than 24 hours), sample count order is arranged so an efficiency check and an instrument background check is made at least every 24 hours while samples are being counted; and/or
- batch QC samples are used to assess instrument performance during the 24 hour period; the efficiency calibration check source used for the efficiency calibration checks, and an instrument blank must be measured in their place.

Continuing calibration verification and background measurements must be within the control ranges produced from their previous measurements:

- If either the QC samples or the instrument efficiency or background check exceed their calculated control limits, the instrument is placed out of service until the source of the out of control condition is identified and corrected; or

- If the failed instrument QC check occurs with a measurement made at the end of measuring a sample preparation batch, then all the samples in the batch are recounted after the source of the out-of-control condition is identified and corrected.

**Finding:** The laboratory does not assess instrument QC at the end of every batch. **This is a repeat finding from the audit performed in July 2008.**

## 8.12 Instrument and Method Performance Charts/Records

### 8.12.1 Requirement – CM Ch. VI, Section 7.8 (Check List Items 9.8.2, 9.8.5, and 9.8.6)

Quality control performance records or control charts should be maintained for each method used by the laboratory for compliance monitoring sample measurements. Laboratory-specific performance warning and control limits for each parameter monitored for both instruments and methods should be recalculated every 20 measurements for each QC parameter. When a

QC result exceeds its calculated control limit:

- All measurements using the associated method or instrument must cease; and
- The source of the out-of-control condition is identified and corrected.

**Finding:** It was observed that the laboratory retired control charts for calendar year 2010 and was collecting data to establish control charts for 2011. However, the control charts were not complete while the laboratory was running compliance samples. The laboratory must maintain control charts for each method until the older control charts can be updated.

### 8.13 Computer programs

#### 8.13.1 Requirement – CM Ch. VI, Section 8.6 (Check List Item 10.8)

Computer programs should be verified initially and periodically by manual calculations, and the calculations should be available for inspection. Access to computer programs and electronic data should be limited to appropriate personnel.

**Finding:** Computer programs are not verified periodically by manual calculations as evident in audit finding 8.1.2. The laboratory must implement a verification and validation of all computer programs that calculate compliance sample reporting results.

## 9.0 Recommendations

The auditor offers the following recommendations to increase the legal defensibility of the data produced by ELI Radiochemistry Laboratory.

### 9.1 Organization and Management – Laboratory Personnel

#### 9.1.1 CM Ch. VI, Section1.2 (Check List Item 1.6)

The **Laboratory Analyst,** at a minimum, should have:

- a bachelor's degree in chemistry or an equivalent degree; and
- one year of experience in the measurement of radioactive analytes in drinking water.

**Recommendations** – Only one of the radiochemistry analysts has a chemistry bachelor's degree. The remaining analysts have high school diplomas; however, by examination of training records, all of the analyst have had broad-based radiochemistry training that was provided by the laboratory's contract Ph.D. Radiochemist. It is highly recommended that the laboratory continue providing radiochemistry training to the analyst to solidify the legal defensibility of radiochemistry produced in the laboratory.

### 9.2 Quality Systems – Laboratory Quality Assurance Plan

#### 9.2.1 CM Ch. III, Section 11.2 (Check List Item 2.3)

The QA Plan addresses the process used to identify clients' Data Quality Objectives (DQOs).

**Recommendations** – The laboratory's QAM states that project specific DQOs must be established for both field and laboratory operations; however, the QAM falls short on detailing the "who-what-when-and where" of how the process takes place. It is recommended that the laboratory expand the discussion, in a QAM revision, of how they identify and document each client's DQOs.

### 9.3 Liquid Scintillation Counting (LSC) system

#### 9.3.1 CM Ch. VI, Section 3.1.1 (Check List Item 6.3.7)

When LSCs are idle, the efficiency calibration and background should be checked weekly to confirm the LSC's ready status for sample measurements.

**Recommendations** – The laboratory does perform prior-to-use efficiency calibration and background checks; however, it is highly recommended that these checks are performed weekly to confirm the LSC's ready status for sample measurements.

### 9.4 Maintenance of Records

#### 9.4.1 CM Ch. VI, Sections 7.6 and 8.1 (Check List Items 9.6.1 and 10.1)

Calibration data and maintenance records on all radiation instruments and analytical balances should be maintained in a permanent record and should be maintained:

- in a permanently bound notebook with all entries made in ink; or
- as an electronic record, with information stored on a computer system that is password protected and backed up weekly with a copy of the backup data stored off site to be considered a permanent record.

Laboratories are to retain sufficient data and documentation for compliance monitoring samples so that their receipt and any measurement made can be reproduced if validation of the data is required.

**Recommendations** – Some of the laboratory records were hard to follow (e.g. gas-flow proportional absorption efficiency curves. It is recommended that the laboratory organize their calibration records in a more efficient manner.

## 10.0 Certification Status

The radiochemistry findings in this report primarily impact documentation of results and quality of the data produced. The Certification Officer **is not recommending certification** for any requested analytes, pending the laboratory's providing an acceptable corrective action plan to address the findings.

### ELI Analytical Methods for Radiochemistry

**Recommendation: Certified (C), Approved (AP), Provisional (P), Not Certified (NC)**

| CONTAMINANT | ON-SITE REVIEW 4/12/2011 | |
|---|---|---|
| | | Method |
| Gross Alpha | NC | EPA 900.0 |
| Gross Beta | NC | EPA 900.0 |
| Radium-226 | NC | EPA 903.0 |
| Radium-228 | NC | Ra-05 |
| Strontium-90 | NC | EPA 905.0 |
| Tritium | NC | EPA 906.0 |
| Uranium | NC | EPA 908.0 |
| Cesium-134 | NC | EPA 901.1 |
| Iodine-131 | NC | EPA 901.1 |
| Gamma Emitters | NC | EPA 901.1 |

## 11.0 SDWA Auditors

[redacted]

May 12, 2011
Date

## 12.0 EPA Certification Officer

[redacted]
Marcie Hall

5-18-11
Date

# On-Site Laboratory Assessment Report (SDWA)

## Radiochemistry

(Final 8/4/2008 LU)

## Report

**Energy Laboratories, Inc.**
**2393 Salt Creek Highway (82601)**
**P.O. Box 3258**
**Casper, WY 82602**

**Audit ID: SDWA-Energy Laboratories-063008**
**June 30–July 1, 2008**

**Prepared by:**
[redacted]
**Environmental Scientist Leader - Senior Radiochemist**
**Computer Sciences Corporation (CSC)**
**6101 Stevenson Avenue**
**Alexandria, VA 22304**

**Prepared in Support of**
**EPA Work Assignment 2-21 under EPA Contract No. EP-C-05-045**

**Inspected by:**
[redacted] **CSC**

**and**

[redacted] **EPA**
**U.S. EPA - Region VIII**
**8TMS-Q**
**1595 Wynkoop St.**
**Denver, CO 80202-1129**

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# 1.0 Introduction

On June 30 – July 1, 2008, an on-site radiochemistry evaluation was conducted of the Energy Laboratories, Inc. (ELI). The laboratory is located at 2393 Salt Creek Highway, Casper, WY 82601. The purpose of this inspection was to determine the capability of the laboratory to perform its mission as it relates to the Safe Drinking Water Act (SDWA) program and the analyses of drinking water samples for radiochemistry analytes as performed by ELI.

The review of radiochemistry was conducted by [redacted], Senior Radiochemist, Computer Sciences Corporation, under contract to the U.S. Environmental Protection Agency's (EPA's) Office of Ground Water and Drinking Water. [redacted], US EPA Certification Officer, US EPA Region VIII, 8TMS-Q, 1595 Wynkoop St., Denver, CO 80202-1129, held the opening and closing sessions and participated in the radiochemistry audit.

References used in this report include the *Manual for Certification of Laboratories Analyzing Drinking Water*, 5th Edition (2005, U.S. EPA), hereafter referred to as the Certification Manual (CM), and the *American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories*, ANSI N42.23-1996, hereafter referred to as ANSI.

This assessment included: review of standard operating procedures (SOPs); review of the Quality System (Quality Assurance Plan); interviews; and data review (raw data to final results for Method Detection Limit studies, demonstration of performance/capability studies, proficiency testing (PT) analyses and SDWA compliance data for September 2004 through June 2008.

**Findings (F),** for EPA Region VIII State SDWA Laboratory assessments, are defined as factual, objective statements that provide evidence of non-conformance with any of the following:

- the Agency's mandatory methods for the applicable programs the laboratory supports (SDWA);
- official Agency mandates and policies, e.g., 40 CFR 141.25, CFR holding times and preservation requirements, and from the CM;
- the laboratory's QA/QC Manual, QA/QC procedures, and SOPs; and documented practices that adversely affect the quality of the data (e.g., supported with actual data from case files).

**Recommendations (R)** may also be contained in the Region VIII Laboratory Assessment reports. These are not findings, but are the technical opinions of the auditor (or assessment team) that are offered to further improve the laboratory's quality system. They are not requirements.

In addition, **General Comments** may also be included, that are a partial listing of innovative or exceptional items the assessment team wanted to highlight (not intended to be an all inclusive listing).

Only findings need to be addressed in the corrective action plan by the laboratory within 30 days of the receipt of the assessment report.

This report uses the Audit Program – Checklist 1, Data Quality for Radiochemical Analysis, Revision 2, August 20, 2007, as the evidentiary documentation of the audit. For full details of the audit along with the findings and recommendations, please refer to the accompanying Audit Checklist.

# 2.0 Personnel

The laboratory was represented by [redacted], Branch Manager; [redacted], Quality Assurance Director; [redacted], Radiochemistry Supervisor; [redacted], Radiochemistry Quality Assurance/Quality

Control; [redacted] Technical Proofing and Data Review.

## 3.0 General Comments

Numerous Quality Control protocols were observed during this on-site inspection, and the laboratory has a Quality Assurance Program Plan. Much data and information were requested prior to the inspection (pre-survey package), and the laboratory was helpful in supplying this information.

The laboratory has a very good sample log in and tracking program/process.

The laboratory has a very good standards traceability process.

## 4.0 Proficiency Testing (PT) Samples

The laboratory has a good PT record (with the exception of PT results for gamma spectroscopy). The laboratory withdrew its request for gamma spectroscopy certification during the audit.

## 5.0 Analytical Method References

The list of parameters in Section 6.0 was assessed during this inspection with the associated methodology cited as follows:

(EPA80) - Prescribed Procedures for Measurement of Radioactivity in Drinking Water, August 1980, EPA-600/4-80-032.
(EPA87) - Radiochemistry Procedures Manual, December 1987, EPA 520/5-84-006
(CM) - Manual for the Certification of Laboratories Analyzing Drinking Water, January 2005, EPA 815-R-05-004.

## 6.0 ELI Analytical Methods for Radiochemistry Information

| Parameter | Method |
|---|---|
| Gross Alpha and Beta | Evaporation (EPA80, 900.0) |
| Radium-226 | Radiochemical (EPA80, 903.0) |
| Radium-228 | Radiochemical (EPA87, Ra-05) |
| Strontium-89 | Radiochemical (EPA80, 905.0) |
| Strontium-90 | Radiochemical (EPA80, 905.0) |

## 7.0 Audit Observations

### 7.1 Personnel

#### 7.1.1 Laboratory Management

The laboratory has sufficiently qualified management to meet the requirements of the CM.

#### 7.1.2 Laboratory Staff

The auditors found the staff to be experienced and knowledgeable in the required aspects of radiochemistry principles, analyses, and data handling. The education, training, experience level, and the laboratory staffing could not be fully assessed due to a training record issue and is detailed in the findings below.

## 7.2 Laboratory Facilities

The laboratory has provisions for maintaining the security of samples. The laboratory is roomy and has adequate lighting at the bench top. Exhaust hoods were in place and appropriate to meet requirements for performing radiochemistry procedures. Hood velocities are monitored by the laboratory. Instruments are grounded, and uninterruptible power supplies are available to all instruments. Chemicals are of appropriate grade. Glassware cleaning follows specific requirements in the methods or general requirements in the CM. The laboratory's chemical hygiene plan is adequate to identify and provide for disposal of chemical wastes produced by the laboratory.

Samples with an emission rate exceeding 0.5 mrem/hr cannot be verified by the laboratory because the laboratory is not assessing all samples entering the laboratory. Provisions are not made to store high dose rate samples away from drinking water samples because dose rates are not being assessed. Standards and reference materials are stored away from samples in a separate area of the laboratory.

The radiation protection and waste program is appropriate for federal and State regulations.

## 7.3 Laboratory Equipment and Instrumentation

Operation of the Gas-flow Proportional Counting Systems generally met the CM requirements.

## 7.4 General Laboratory Practices

Laboratory operational practices generally meet the CM requirements.

## 7.5 Analytical Methodology

Standards' National Institute of Standards and Technology (NIST)-traceable preparations meet CM requirements.

The approved radioanalytical methods cited in 40 CFR 141.25(a) and (b), Table VI-1 are modified for use in the radiochemistry laboratory without the modifications detailed in the SOPs. The laboratory does not have alternate test procedure (ATP) approvals for these procedure modifications. Details of these modifications are noted below.

## 7.6 Sample Collection, Handling, and Preservation

Samples are submitted to the laboratory, for analysis, from various clients from across the United States. Handling, compositing, and preservation of samples do not meet CM requirements in all cases, and these deficiencies are detailed below.

## 7.7 Quality Assurance

The laboratory has a written, separately prepared QA plan (QAP) and in general a very good overall program, with exceptions noted below. Laboratory safety plans were thorough.

## 7.8 Records and Data Reporting

Records and Data Reporting generally met CM requirements, with exceptions noted below.

# 8.0 Audit Findings

## 8.1 Organization and Management

### 8.1.1 Laboratory Personnel

**Requirement – CM Ch. VI, Section 1.2; Checklist Items 1.6, 1.7, and 1.8**

If the analyst is responsible for the operation of analytical instrumentation, he or she is required to have:

- completed specialized training offered by the manufacturer, another qualified training facility, or
- served a period of apprenticeship under an experienced analyst and the duration of this apprenticeship is proportional to the sophistication of the instrument.

Completion of this apprenticeship period for instrumentation should be documented and maintained in a training file.

**Finding** – Analyst training could not be assessed because staff training records were taken by another agency and the records were not available for review. The auditor considers this a serious finding.

### 8.1.2 Laboratory Safety

**Requirement – CM Ch. VI, Section 4.4; Checklist Item 2.16**

Guidelines in the Laboratory Safety Manual, the Chemical Hygiene Plan, or the SOPs should include:

- safety training and protection information specific to a radiochemistry laboratory;
- operations used by the laboratory in handling hazardous materials;
- identification of hazardous materials used by the laboratory;
- health risks that are possible if someone is exposed to the hazardous materials;
- precautions that workers should take to protect themselves from exposure and possible injury;
- when and how radiation shielding is used to protect analysts and technicians from harmful levels of radioactivity;
- description of circumstances that warrant the use of protective equipment;
- the use of gloves, laboratory coats, eye protection and appropriate pipetting techniques to avoid exposure and possible injury from chemicals and radioactive substances; and
- documentation of relevant training.

**Finding** – During the course of the audit, it was observed that the laboratory staff members were drinking at their analytical work stations. This practice is contrary to ELI's Safety Manual and is not in accordance with good laboratory practices.

### 8.1.3 Initial and Ongoing Demonstrations of Proficiency for Analysts and Technicians

**Requirement – CM Ch. VI, Section 1.5; Checklist Items 3.3 through 3.5**

All analysts and technicians must demonstrate the ability to conduct measurements with acceptable accuracy, precision, and freedom from interferences and demonstrate this proficiency:

- before beginning the analysis of compliance samples; and annually
- bench sheets and instrument printouts made during these demonstrations of proficiency should be retained and be available for inspection

Demonstrations of Proficiency must be done by conducting an MDL study as described in 40 CFR part 136, Appendix B, or by the following the alternate procedure listed in Checklist Item Number 3.3.

Demonstrations of Proficiency is performed by:

- preparing and measuring a sample set of at least four reagent blanks and four laboratory fortified blanks that have the radioanalyte of interest added to them at a known concentration appropriate for the relevant method;
- the activity level added to the laboratory fortified blanks should be between the radioanalyte MCL and its required detection limit;
- the mean recoveries and the standard deviation of the recoveries of the replicate measurements should be consistent with the requirements for accuracy and precision described in CM Ch. VI, Section 7.7;
- reagent blank measurements have a mean result below the method detection limit for each analyte measured; and
- all Initial and Ongoing Demonstrations of Proficiency, along with all records related to methods training and external training relevant to laboratory operations, should be recorded in a training file specific for each analyst and technician.

Data produced by analysts and technicians who have not completed their training, or who do not have a current demonstration of proficiency on record, as well as instrument operators still in the process of obtaining the required training or experience, are acceptable only when:

- the data are reviewed and validated by a fully qualified analyst or the laboratory supervisor; and
- the fully qualified analyst or the laboratory supervisor provides a permanent record of their review and the data's acceptability by placing their signature and date in ink on any bench sheets, calculation sheets, or reports generated.

**Finding** – Analyst training could not be assessed because staff training records were taken by another agency and the records were not available for review. The auditor considers this a serious finding.

## 8.2 Laboratory Facilities

### 8.2.1 Requirement – CM Ch. VI, Section 2.1 (Check List Item 4.6)

Any sample having an emission rate in excess of 0.5 mrem/hr must be stored in a secured location away from drinking water samples.

**Finding:** The laboratory does not monitor all incoming samples to satisfy this requirement; thus can not insure cross contamination potentials are identified and separated from drinking water samples.

## 8.3 Analytical Methods – Standard Operating Procedures

### 8.3.1 Requirement – CM Ch. VI, Section 5.1 (Check List Item 7.2.5)

Laboratory's SOP for **Radium 228** (Radiochemical) is consistent with the methods listed in 40 CFR part 141.25(a) and (b), Table VI-1, and any EPA-approved modifications **should** be noted.

**Finding:** The laboratory is seeking Ra-228 certification by EPA approved method Ra-05. However, a review of the laboratory's SOP revealed that the SOP combines sections from EPA method 903.0 and Ra-05, which is inconsistent with the approved Ra-05. Modifications are not specifically called out, nor does the laboratory have an ATP approval for this SOP. It is recommended that the laboratory use the EPA-approved Ra-05 method, to analyze for Ra-228. The laboratory may elect to use EPA method 903.0, and should submit acceptable PT results for this method; also, adjust their certification request accordingly. The laboratory may apply for ATP approval of their hybrid method. It should be noted that the laboratory is passing performance testing samples with their SOP; however, it is incumbent on the laboratory to demonstrate the alternate

method's performance (method validation).

## 8.4 Sample Collection, Handling, and Preservation

### 8.4.1 Requirement – CM Ch. VI, Section 6 (Check List Items 8.3 and 8.4)

Sample preservatives provided by the laboratory should be screened for radioactive content by lot number prior to their use in the laboratory, and the results documented. Samples preserved with reagents that are not provided by the laboratory are to be accompanied by a radioactive-free field blank sample that is preserved in the same manner as the submitted sample.

**Finding:** The laboratory is performing reagent checks only with method blanks. The laboratory does not know if acids used for preservatives (and also method reagent preparation) are free of radioactivity until after-the-fact analysis. It is recommended that the laboratory implement a preservative screening program to avoid artificially biasing compliance drinking water samples. Also, the laboratory does not require samplers, who preserve samples with reagents not provided by the laboratory, to provide a field blank sample in order to assess if any radioactivity has been added, to the compliance drinking water sample, from the preservatives used by the sampler(s).

### 8.4.2 Requirement – CM Ch. VI, Section 6.1 (Check List Item 8.8)

A sample of the preservative should accompany the composited sample to the laboratory to determine the contribution of radioactivity, if any, from the addition of the preservative.

**Finding:** The laboratory does not require samplers, who preserve samples with reagents not provided by the laboratory, to provide a field blank sample in order to assess if any radioactivity has been added to the compliance drinking water sample from the preservatives used by the sampler(s).

## 8.5 Radiochemistry Quality Assurance

### 8.5.1 Requirement – CM Ch. VI, Section 7.2 (Check List Item 9.2.2)

Balances must be re-calibrated at least annually with ASTM Type I weights.

**Finding:** The laboratory has new balances in use that do not have documentation of calibration.

### 8.5.2 Requirement – CM Ch. VI, Section 7.7.1 (Check List Items 9.7.5 through 9.7.7)

An RPD result that exceeds its calculated control limit (which ideally should be 20 percent or less) indicates the precision of the sample preparation batch is questionable, and data reported from these results should be flagged as having questionable precision. Duplicated sample measurement activities that are less than 5 times the radioanalyte's detection limit, and exceed 20 percent RPD when compared to the first measurement for the sample are:

- re-evaluated using the two measurement's replicate error ratio (RER) using the formula:

$RER = |A - B| / SQRT( s_a^2 + s_b^2 ) \leq 2$

- If the RER exceeds 2, it is noted as unacceptable

If precision assessments exceed their limits (RPD or RER):

- Calculations and procedures are examined and samples recounted and re-evaluated.
- If the precision assessments are still unsatisfactory after the samples are recounted, then all sample results in the preparation batch are to be reported with a qualifier to indicate the measurement has questionable precision.
- If the client requires unqualified results, then all the samples in the sample preparation batch are

discarded and then re-measured using new aliquots of the sample if holding times allow and sufficient volume is available. Otherwise, the water is resampled and reanalyzed.

**Finding:** Some of the laboratory's SOPs require these criteria and others state ±30 percent. The laboratory LIMS is monitoring all methods at ±30 percent. The laboratory needs to change their LIMS criteria to reflect the CM requirements and revise their SOPs to be consistent with this requirement. DER is not addressed in the laboratory's SOP for Ra-226 (ELI-C-50-EPA-903.0). The laboratory needs to review and revise their SOPs to conform to this CM requirement. The laboratory's LIMS needs to be reset to monitor RPD and RER in accordance with the CM requirements cited above.

### 8.5.3 Requirement – CM Ch. VI, Section 7.7.1 (Check List Items 9.7.9 and 9.7.11)

MS performance is assessed using the percent recovery of the known activity of radioanalyte added to the sample and **should** be within:

- ± 20 percent of the amount of activity added; or
- for gross alpha, gross beta, and Ra-228, **should** be within ± 30 percent of the amount of activity added.

When MS assessments of accuracy exceed their calculated control limits,

- calculations and procedures **should** be (are) examined and samples recounted.
- If the assessment is still unsatisfactory after the samples are recounted, then all sample results in the batch **should** be flagged as possibly biased low or high (as the result indicates) due to matrix effects.

**Finding:** The laboratory LIMS is monitoring all methods at ±30 percent. The laboratory needs to change their LIMS to reflect the requirements of this line of inquiry and revise their SOPs to be consistent with this requirement. However, it is noted that the laboratory is recounting samples if they exceed their current limit of ±30 percent.

### 8.5.4 Requirement – CM Ch. VI, Section 7.7.3 (Check List Items 9.7.13 through 9.7.15)

Assessing the preparation batch accuracy independent of matrix effects using laboratory fortified blanks (LFB):

- LFBs are prepared using deionized water;
- an LFB shall be prepared with each preparation batch of samples;
- LFBs are processed through the method along with the sample batch; and
- LFBs are assessed using percent recovery calculations and **should** be within the control limits ± 10 percent of the amount of activity added to the LFB sample;

Except:

for gross alpha, gross beta, and Ra-228, LFBs **should** be within ± 20 percent of the amount of activity added

If LFB assessments exceed their limits, calculations and procedures **should** (are) be examined and samples recounted.

If the LFB assessment is still unsatisfactory after the samples are recounted:

- the batch is be considered contaminated;
- all samples in the sample preparation batch are discarded;
- the source of contamination is identified and addressed; and
- new aliquots of the sample(s) in the preparation batch are measured if holding times allow and sufficient volume is available.

**Finding:** The laboratory LIMS is monitoring all methods LFBs at ±30 percent. The laboratory needs to change

their LIMS to reflect the requirements of this line of inquiry and revise their SOPs to be consistent with this requirement.

### 8.5.5 Requirement – CM Ch. VI, Section 7.7.3 (Check List Item 9.7.16)

Assessing instrument drift during sample measurements.
Efficiency and background are checked:

- before measuring compliance monitoring samples; and
- after measuring compliance monitoring samples; and
- during long analytical batch counting times (greater than 24 hours), sample count order is arranged so an efficiency check and an instrument background check is made at least every 24 hours while samples are being counted; and/or
- batch QC samples are used to assess instrument performance during the 24 hour period; the efficiency calibration check source used for the efficiency calibration checks, and an instrument blank must be measured in their place.

**Finding:** The laboratory is not assessing instrument drift during sample measurements by checking efficiency and background after measuring compliance monitoring samples.

## 9.0 Recommendations

The auditors found ELI as a very professional operation, with legally defendable data for compliance monitoring drinking water samples, and cannot offer any additional recommendations other than correcting the audit findings cited above.

## Certification Status

The radiochemistry findings in this report primarily impact documentation of results and in no case was the quality of the data produced impacted. The COs are recommending full certification for all requested analytes, pending the laboratory's providing an acceptable corrective action plan to address the findings.

### ELI Analytical Methods for Radiochemistry

**Recommendation: Certified (C), Approved (AP), Provisional (P), Not Certified (NC)**

| CONTAMINANT | ON-SITE REVIEW 6/30/2008 | |
|---|---|---|
| | | Method |
| Gross Alpha | C | EPA 900.0 |
| Gross Beta | C | EPA 900.0 |
| Radium-226 | C | EPA 903.0 |
| Radium-228 | C | EPA Ra-05 |
| Radioactive Strontium-89 | C | EPA 905.0 |
| Radioactive Strontium-90 | C | EPA 905.0 |

10.0 SDWA Auditors

[redacted]

6/4/08
Date

Jim Gindelberger

Date